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HEXABROMOCYCLODODECANE, TETRABROMOBISPHENOL-A AND INORGANIC ADDITIVES IN POLYMERS FROM ELECTRIC AND ELECTRONIC WASTE

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SUMMARY: The environmental aspects of the use of flame retardants (FRs) in e-waste and their recycling are of special concern. The widespread use of pigments, stabilizers and FRs in consumer housing plastics imposes environmental and economic impacts in the process chain as contaminants could limit the recovery of polymer and reduce post-recycled value. Presently, there is a paucity of information on the occurrence of the most widespread halogenated organic compounds, namely tetrabromobisphenol-A (TBBPA) and hexabromocyclododecane (HBCD) and the composition of inorganic substances in polymeric compounds. To provide quantitative data of target compounds in polymeric articles, this study focuses on the content levels of TBBPA and HBCD stereoisomers (α-, β-, and γ-), as well as of selected metals. A number of obsolete multi-functional printers and personal computer mainframes were subjected to microwave-assisted chemical extraction and then leachates were analysed. Detection levels revealed that the amount of TBBPA and HBCD varies significant even among plastics of the same category with highest values observed in a sample marked as a blend of polycarbonate and acrylonitrile-butadiene-styrene polymer (PC+ABS, up to 146,838 μg TBBPA/kg) and one made of ABS (up to 125 μg ∑HBCD/kg) from printers' samples. Concentration mean values for elements used as fillers such as Ca, Mg, Sb and Al were found elevated in "ABS", "ABS-FR, PVC" and "ABS-FR(40)", respectively. Hence, such data would serve to discuss appropriate recycling process scenarios and risk assessment of potential hazards.

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1. INTRODUCTION

Due to their relatively inexpensive production, durability, physical and chemical properties, the use of plastics has increased in electric and electronic equipment (EEE), accounting for about 20-30 wt% of the total amount (Arends et al., 2015; Ma et al., 2016). Although plastics have a high recycling potential from a material recovery perspective; challenges have risen due to the diverse polymers types (Covaci et al., 2009), inorganic or organic additives, such as Pb, Sb, Hg, Cd, Ni, Sb added as stabilizers or pigments (TiO_2, ZnO, Fe_2O_3 etc.) and mainly, flame retardants (FRs) (Martihno et al., Yu et al., 2017). Latters are presented in the most of the plastic components used in electronic products, which may involve future environmental risks. In principle, FRs act by interrupting the process of combustion in the solid and liquid phases of the substrate or in the gas phase (Dufton, 2003). It is noteworthy to be mentioned that their efficiency is often enhanced by the addition of inorganic FRs (Bisschoff, 2000) by forming a crust on the polymer surface during thermal stress that prevent oxygen to access (Puype et al., 2015). Also, fillers like calcium carbonate, kaolin, talcum powder etc. further enhance polymers' strength, toughness and reduce the production cost (Tang et al., 2002). Another advantage of using inorganic fillers is that decompose endothermically with the release of inert gases or vapour, enhancing the FR effect (Hull et al., 2011).

Halogenated FRs are primary based on chlorine (chlorinated flame retardants (CFRs)) or bromine (brominated flame retardants (BFRs)) content, with the last ones to be about two thirds more effective in terms of thermal stability (Bisschoff, 2000). BFRs such as polybrominated diphenyl ethers (PBDEs), tetrabromobisphenol A (TBBPA) and hexabromocyclododecane (HBCD), are the most widespread halogenated organic compounds that in general added to polymers used in EEE (Someya et al., 2016). Amongst them, HBCD has been designated as persistent organic pollutants (POPs) by the Stockholm Convention (Haffner & Schecter, 2014) and has been included in the list of proposed restricted substances (RoHS Directive) (Zhong et al., 2010). Several studies have reported HBCD as potentially bioaccumulative and exerting endocrine-disrupting effects on organisms (Kemmlein et al., 2009). Based on *in vivo* experiments, TBBPA has upgraded to group 2A depicting carcinogenic potential to humans, by International Agency for Research on Cancer (IARC) (Grosse et al., 2016). Broadly speaking, BFRs always co-exist with metals in electronic products (Zhong et al., 2010). Aluminum hydroxide and magnesium hydroxide compounds are used as halogen-free alternatives to BFRs, while antimony trioxide, zinc borate, and certain molybdenum compounds are used as synergists mixed with different types of FRs (Marris et al., 2015).

Table 1. Elements related to the identification of inorganic FRs and fillers (Maris et al., 2015; Weber & Kuch, 2003).

Element	Flame retardants	Mineral Fillers
Mg	Magnesium hydroxide	Magnesium silicate
Al	Aluminum trihydroxide	Aluminum silicate
Si	-	Glass fibers
Ca	-	Calcium carbonate
Ti	-	Titanium oxide
Sb	Antimony oxide	-
Zn	-	Nano zinc oxide
Fe	Ferric oxide	-
Sn	Stannic oxide	

The most widely practiced methods for recycling plastics are mechanical recycling (manual sorting and floatation etc.) and chemical recycling (Zhao et al., 2018). Even though, the recyclability of polymer flame retardant may be restricted due to the possibility of the formation of polyhalogenated dioxins and furans (PCBB/PBDD and PCDF/PBDF) and other organic halogen compounds

(PXDD/PXDF) during end-of-life management process (Barontini et al., 2014; Beccagutti et al., 2016; Taurino et al., 2010). The total amount of dioxins and furans formed during thermal processes depends largely on the quality of precursor compounds (halogen content) and the specific conditions of the treatment. To the purpose of improving the operation of recycling processes, avoiding the primary sources' depletion, decreasing CO_2 emissions and saving energy (Zhao et al., 2018), identification and sorting of samples according to the FR type and polymer type is necessary prior to recycling process to avoid environmental emissions and increase the value of the post-recycling plastic.

Therefore, a systemic approach should be proposed to determine the occurrence of such chemicals in end-of-life EEE in such a way that could improve recovery and eliminate the toxic emissions. Currently, there are only a few studies regarding the quantitative analysis of the most common BFRs in e-waste plastics. Taking into account the need for greater knowledge of WEEE plastic content, this research focuses on the presence of selected BFRs in relation with inorganic additives in multi-functional printers and computer housings. This involves quantification of TBBPA and diastereomers α-HBCD, β-HBCD and γ-HBCD as well as the determination of the actual concentration levels of target metals and metalloids.

2. MATERIALS AND METHODS

For the purpose of the study, a total of 16 plastic housings from post-consumer devices have been investigated in regard to the addition of certain BFRs and their metal composition. Particularly, 13 samples from multi-functional printers and 3 samples from personal computer mainframes were selected to undergo acid digestion and solvent extraction under microwave irradiation for the quantification of metals and metalloids, TBBPA and total HBCD (sum of α-HBCD, β-HBCD and γ-HBCD) content level.

Initially, the plastic parts obtained were examined to assess the polymer type. This information was provided by the moulding mark declaring manufacturer technical documents on the polymer type and chemical composition of the FRs in each plastic casing. Note that based on EN ISO 11469:2016, which specifies a system of uniform marking, all products fabricated from plastic materials are marked. In general, the provided identification intends to contribute to safe handling, waste recovery or proper disposal of this waste stream. In this research, the majority of plastic samples examined, was made from the copolymer ABS (acrylonitrile-butadiene-styrene) and PC (polycarbonate) blend (n=9). Specifically, the plastic articles identified with the symbol ">PC+ABS-FR(40)<" refers to the abbreviated term of PC and ABS blend with a halogen-free organic phosphorous compound as a flame retardant. In the certain blend, the main component first illustrated (PC). Following by number (n=5), pure ABS plastic parts with halogen-free organic phosphorous compound addition were marked with the abbreviation term ">ABS-FR(40)<". Only 2 labels were indicated that the plastic parts were made of blends of two polymer types, either ABS with high impact polystyrene (HIPS) or ABS with polyvinyl chloride (PVC) (Table 2). The symbol ">ABS-FR(17)<" denotes that the polymer type is ABS blend with aromatic brominated compounds in combination with antimony compounds as a synergistic inorganic flame retardant.

For the experimental procedure, the plastic components, at first, manually chopped into foils to a size of less than 2 x 1 mm using hand cutting tools; then, the foils were embattled with liquid nitrogen to further reduce the particle size (less than 1 mm). The cryogenic grinding may facilitate the solid-liquid extraction by increasing the surface contact of the sample. Afterwards, the samples were stored in the dark at a temperature of 25±2 ^{o}C to prevent degradation phenomena on polymer articles (Yousif and Haddad, 2013). For the extraction of TBBPA and HBCDs, microwave-assisted extraction method

(MAE) was employed. The MAE procedure was carried out for approximately 1 g of solid sample and addition of 20 mL of isopropanol/*n*-hexane (IP/n-hexane, 1:1, v/v) into the closed vessels designed for microwave sample preparation system (Multiwave 3000, Anton Paar, Graz, Austria). The produced extracts were subjected to centrifugation at 2,500 rpm for 5 min. After decanting the liquid phase, a mixture of deuterated $^{13}C_{12}$-α-HBCD and $^{13}C_{12}$-TBBPA (Wellington Laboratories, Guelph, Canada) was introduced in the vial. In order to reduce the volume to 1.5 mL, a water-bath rotary evaporator (Rotavapor R-205, Büchi, Switzerland) was operated at 40 °C. The collected extracts were filtered using mini uniprep 0.2 μm pore polypropylene (PP) filter vials (Whatman, UK) before analysis with a high-performance liquid chromatographer (HPLC) coupled to coupled to a quadrupole time-of-flight mass spectrometer (QTOF/MS). The detection limit for TBBPA was 5 ng/g and for α-HBDC, β-HBDC and γ-HBDC isomers was 3, 10, 20 ng/g, respectively.

Determination of polymeric samples composition takes place in Mars 6 Microwave Reactor System by CEM Corporation aiming at subsequent digested leachates analysis. Two digested samples (duplicate experiment) were analysed to ensure repeatability of the results and examine any experimental error. With respect to digestion conditions, approximately a quantity of 250 mg of each polymer sample is placed in PTFE vessel by adding 10 mL nitric acid (HNO_3, Sigma-Aldrich 65%). The method was set to heat to a temperature of 210 °C for 15 min with a ramping time 25 min and allowed to cool to room temperature before venting and transferring to an appropriate container.

Reagent blanks were also taken through the entire procedure. Thereafter, all solutions were filtered with PTFE syringe filter 25 mm of a pore size 0.45 μm (Lab Solutions, Greece) in vials and then diluted with deionized water to a total volume of 45 mL. The subsequent chemical analysis for the determination of toxic metals in all samples was performed using an Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Agilent 7500cx) equipped with an Auto-sampler Series 3000. The chemical analyses focused both on toxic metals referred to the Directive 2002/95/EC, cadmium (Cd), lead (Pb), chromium (Cr), arsenic (As), zinc (Zn) as well as on other most commonly found inorganic additives on EEE even derived from the incorporation of inorganic FRs compounds or pigments namely, nickel (Ni), titanium (Ti), calcium (Ca), sodium (Na), iron (Fe), antimony (Sb), silicon (Si), aluminum (Al), magnesium (Mg) and tin (Sn).

3. RESULTS AND DISCUSSION

3.1. Target BFRs content value in e-waste plastics

The details of the individual polymeric e-waste samples, the measured TBBPA and HBCDs values are presented in Table 2. From the 16 samples in total, TBBPA and HBCD could be detected in 13 samples. The results show that concentration levels of TBBPA vary significantly among the samples, even if it refers to the same category of waste, ranging from values below the detection limit (BDL) to the maximum value of 146,838 μg/kg, while the mean value was calculated at about 12,581 μg/kg. As illustrated, TBBPA was not detected in plastic parts marked as pure ABS copolymer. The respective mean value of total HBCD (∑HBCD) was calculated about 39 μg/kg. To be noted, when calculating averages samples below the detection limit were assumed to be equal to zero.

The presence of TBBPA in household electronic appliances (desktop mainframes, monitors covers etc.) has been proved in previous studies in an average value of 1,780 μg/kg and a range of 600 to 2,430 μg/kg, (Yu et al., 2017). Similarly, in personal computers boards, Kajiwara et al. (2011) identified about 800-2700 μg TBBPA/kg. Overall, from previous studies, it was conducted that even TBBPA was the most frequently and abundantly detected BFR, significant variations observed. Particularly, in the research campaign of Gallen et al. (2014), TBBPA in computer devices was detected in the 50% of the samples with the highest value of 150,000 μg/kg, and conversely, electric

accessories such as powerboards and adaptors' had greater amounts of TBBPA, up to 164,000 mg/kg. Compared those results to computer housing content values of other studies, the detection levels have showed lower enrichment capacities of TBBPA (15,804 mg/kg). Those variations could be probably attributed to the different composition of e-plastic components, the role of each consumer device and/or be associated with additional or other substitute compounds used also as FRs.

Another explanation lies on the fact that an amount of flame retardants may be decomposed during the period of the electrical and electronic equipment used in relation with the operating temperature and sunlight irradiation (Chen et al., 2012). Hence, a systematic approach is rather difficult and complex due to the heterogeneity of the consumer products.

Table 2. Detailed sample description, concentration (TBBPA and HBCD in μg/kg) in polymeric waste plastics (1-13 multi-functional printers; 14-16 personal computers desktop).

		Polymer type	Tetrabromo-bisphenol A (TBBPA)	Hexabromocyclodecanes (HBCDs) (μg/kg)			Total hexabromocyclodecane (∑HBCD)
				α-	β-	γ-	
Limit of detection (ng/g)			5	3	10	20	
Sample number	1	PC+ABS-FR(40)	86	10	BDL	BDL	10
	2	ABS-FR(40)	133	17	39	BDL	56
	3	PC+ABS-FR(40) GE Resin HP P/N	68	BDL	14	BDL	14
	4	PC+ABS-FR(40)	85	BDL	BDL	BDL	BDL
	5	PC+ABS-FR(40)	BDL	BDL	BDL	BDL	BDL
	6	ABS-FR(40), ABS, PC+ABS FR(40)	770	17	47	BDL	64
	7	ABS-FR(40)	9,503	10	17	BDL	27
	8	ABS-FR(40)	448	63	44	BDL	107
	9	PC+ABS-FR(40)	138	BDL	BDL	104	104
	10	ABS	BDL	18	97	10	125
	11	PC+ABS-FR(40)/ ABS-FR(17)	146,838	10	10	BDL	20
	12	ABS	BDL	BDL	17	BDL	17
	13	ABS, HIPS	33,860	BDL	10	BDL	10
	14	PC+ABS-FR(40)	120	BDL	BDL	BDL	BDL
	15	ABS, PC+ABS-FR(40)	6,336	10	BDL	15	25
	16	ABS-FR, PVC	2,914	BDL	BDL	38	38

* BDL refers to the values that are below the detection limit of the analysis method.

The use of HBCD as the second most prevalent flame retardant has been documented previously, too (Gallen et al., 2014; Zhong et al., 2010). Among the samples analysed, ∑HBCD was found in concentrations ranging from 10 to 125 (μg/kg). The highest concentrations were associated with samples of PC and ABS blends (104 μg/kg), ABS-FR(40) (107 μg/kg) and ABS (125 μg/kg) derived from multi-functional printers articles. Although, those values are lower than the range of <0.0003 – 1600 mg/kg quantified in polymers of consumer e-waste housings according to similar studies (Drage et al., 2018).

Based on the detection level of the individual isomers, α-, β- and γ-, it has shown that the β-HBCD is the main isomer, followed by α-HBCD. However, this isomer distribution is different for the most of the polymeric samples analysed (10 - 97 μg β-HBCD/kg). Previously, it was mentioned by Kajiwara et al. (2011) that HBCDs were considered minor BFRs in the e-waste components

examined, with the α-diastomereomer concluded to be the most commonly used in electronic appliances. Moreover, according to the results of several studies, multiple BFRs were found even in the same polymer type, whilst a small proportion of HBCD has been used to treat electronic equipment (Drage et al., 2018).

From another point of view, information on the occurrence of HBCDs in EEE devices are limited with the target compound to be reported as total HBCD (Abdallah et al., 2017; Drage et al., 2018). Although, in order to provide a toxicology profile of the compound, the occurrence of HBCD in biological matrices, especially α- isomer or in environmental samples, mainly γ- one (Daso et al., 2017), screening of the different isomers was considered more often.

3.2. Inorganic composition of e-waste plastics

In general, the acid digestion results have shown very low or non-detectable amounts of metals of toxicological concern, such as As, Cd, Ni, total Cr and Pb. These results can be compared with other studies presenting similarities in the Pb, Cd and Cr (Dimitrakakis et al., 2009). In Figure 1 the total amount of each of the examined metal (mg/kg) in the polymer samples, is displayed, providing maximum, minimum and mean values as well as 25% and 75% percentiles. In this research, the inorganic FRs were characterized based on associated molecules. For instance, Maris et al. (2015) have underlined that filers in polymer matrices, like talc, are characterized by the existence of magnesium and silicon elements or aluminum silicate (known as kaolin) by aluminum and silicon, severally. In the same manner, also other compounds might be related to the identification of inorganic FRs and fillers in plastics, generally (Maris et al., 2015).

The Mg content was found relatively higher in the ABS samples (258.03±2.67 and 281.29±2.67 mg/kg, respectively) followed by the total content measured in samples marked as PC+ABS-FR(40) (range 147.63 to 147.63 mg/kg). Broadly, the magnesium content may be attributed to the presence of talc ($Mg_3Si_4O_{10}(OH)_2$) which is a common additive in polymeric materials (Stenvall et al., 2013). In contrast, in the polymers indicated as a blend of ABS with FR(40), the lowest values of Mg were observed (12.93±0.92 to 24.96±1.35 mg/kg), depicting addition of other inorganic FRs. In particular, in the aforementioned samples, the values of Al was detected in higher levels than Mg ranging from 210.68±1.98 to 120.12±2.08 mg/kg, meaning that aluminium hydroxide ($Al(OH)_3$) has been incorporated in the polymers as a pigment (Hull et al., 2011). Alumina Tri-Hydrate (ATH) or hydroxide and magnesium hydroxide are acid- and halogen-free FR compounds for various plastics, according to Walter et al. (2006).

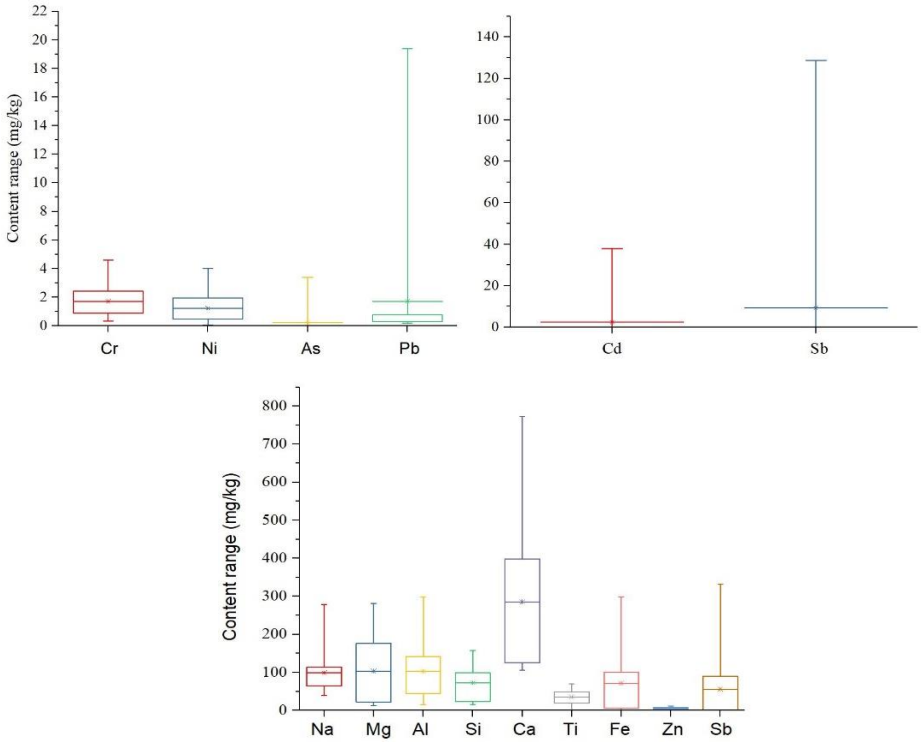

Figure 1. Mean, maximum, minimum values as well as 25% and 75% percentiles of target elements in plastic matrices (mg/kg).

Similarly, the highest content of Ca (773.05±5.95 mg/kg) that was detected in pure ABS sample (n. 7), denotes that ABS polymer is usually filled with inorganic particles like calcium carbonate ($CaCO_3$) (Tang et al., 2002). Additionally, mean values of Si in the plastic examined have calculated about 72.36 mg/kg, whereas Ti content value was found equal to 35.65 mg/kg.

Regarding Sb, variations in values were observed, too. Concentration for Sb obtained for the plastic marked with the description of ">ABS-FR, PVC<" was measured up to 128.74±1.61 mg/kg. Since antimony trioxide (Sb_2O_3) is synergist of BFRs (Schlummer et al., 2007; Vehlow et al., 2000) and organotin PVC stabilisers (Schlummer et al., 2007), the high content could be explained in the certain polymer type. TBBPA content levels in the latter was measured in the highest level of 146,838 μg/kg. Practically the Sb_2O_3 is incorporated in polymers for decreasing the level of the total required BFR for flammability standard (Puype et al., 2015).

4. CONCLUSIONS

In contrast with the high content of plastic fraction in e-waste, recycling process remains challenging due to the lack of sufficient identification and sorting techniques of the polymer types and FRs content. The main aim of this study was to investigate the presence of the most frequency used BFRs, TBBPA and HBCD (sum of isomers α-HBCD, β-HBCD and γ-HBCD) and certain inorganic additives in plastic fractions derived from multi-functional printers and computer desktop mainframes. The articles were selected to undergo chemical treatment under microwave irradiation with a solvent media (isopropanol/ n-hexane) for the extraction of TBBPA and HBCD. Detection levels revealed that the content of BFRs varies from plastic to plastic even among the same waste category. Moreover, to the scope of the present study, a microwave oven system was used for the quantification of the metal content of plastic extracts. Concentration range for elements of concern such as As, Cd, Ni, total Cr and Pb was measured in lower quantity whereas Ca and Mg content in ABS was the highest

denoting the addition of magnesium hydroxide as FR and calcium carbonate as filler. Similarly, Sb concentration was found relatively high in "ABS-FR, PVC" and Al in "ABS-FR(40)" types of plastics. The widespread use of pigments, stabilizers and FRs in consumer plastic imposes environmental and economic effect in the process chain as contaminants could limit the recovery of polymer and reduce post-recycled value. Hence, future work shall be directed towards a systematic screening concerning the material composition. Such data would serve to discuss appropriate recycling process scenarios and risk assessment of potential hazards. Migration possibility of hazardous substances through e-waste recycling processes in exit streams would also be expedient.

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